Name of Procedure:

Extractions and Separations
Extraction of Organic Bases

Suggested Uses:

This is a general procedure used to isolate and purify basic compounds (alkaloids) for further analysis.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:
Fume Hood
Heat source
Eye protection
Laboratory coat
Gloves
Beaker(s), sample vial(s) or other glass container(s)
Filter Paper
Funnel
Glass stirring rod
Graduated cylinder
Pipettes with bulb
Reagent or stock bottle(s)
pH Test paper
Separatory funnel (optional)
Spatula
Test tube(s)
Water

Suitable acid for example:
Hydrochloric acid (concentrated) suggested

Suitable organic solvent(s) for example:
Ethyl ether, Chloroform, Hexane, Methylene chloride, Isopropanol

Suitable base for example:
Sodium hydroxide, Sodium bicarbonate, Ammonia

Drying agent (optional) for example:
Sodium sulfate (anhydrous), Magnesium sulfate (anhydrous)

Formula for Preparing Reagents:

0.6N Hydrochloric Acid Reagent - The reagent may be prepared in any amount provided that the component ratios are kept constant.

1. Measure 95 milliliters of water in a 100 milliliter graduated cylinder.
2. Bring to total volume (100ml) with concentrated Hydrochloric acid. (SAFETY NOTE: Always add the acid to the water.)
3. Pour into a reagent or stock bottle.
4. Properly label bottle.

Ethyl Ether saturated with Hydrochloric Acid Reagent - The reagent may be prepared in any amount provided that the component ratios are kept constant.

1. In a test tube or other glass container, mix Ethyl ether and concentrated Hydrochloric acid in an approximate 1:5 (HCl:Ethyl ether) ratio.
2. Gently shake to mix the layers.
3. Allow layers to separate and remove Ethyl ether for use.

Concentrated Sodium Hydroxide Reagent - The reagent may be prepared in any amount provided that the component ratios are kept constant.

1. Add desired amount of water to beaker or other glass container.
2. Add Sodium hydroxide pellets with stirring until solution is saturated. (No more pellets will dissolve.)
3. Pour solution into a reagent or stock bottle.
4. Properly label bottle.

Expiration Date of Reagents:

0.6N Hydrochloric acid reagent and the concentrated Sodium Hydroxide reagents can be used until depleted provided they are stored in airtight reagent or stock bottles.

Ethyl ether saturated with Hydrochloric acid reagent is prepared fresh as needed.

Application of Procedure on Evidence:

1. Dissolve the sample in the 0.6N Hydrochloric acid reagent or other suitable acid. Check the pH with test paper, if needed, to assure solution is acidic.

2. Extract the acid solution with a suitable organic solvent. Suitable solvents could be but are not limited to: ethyl ether, chloroform, hexane, methylene chloride, 3:1 chloroform/isopropanol. The chemical properties of the organic base being extracted and the chemical properties of the other substances mixed with the organic base being extracted will determine which organic solvent is used.

3. Discard solvent washings or retain for further analysis, as needed.

4. Make the acidic solution basic by adding concentrated sodium hydroxide solution, sodium bicarbonate, or other suitable base. The chemical properties of the organic base being extracted and the chemical properties of the other substances mixed with the organic base being extracted will determine which base is used. Check the pH of the solution with test paper, if needed, to ensure solution is basic.

5. Extract the basic solution with a suitable organic solvent. Suitable solvents could be but are not limited to: ethyl ether, chloroform, hexane, methylene chloride, 3:1 chloroform/isopropanol. The chemical properties of the organic base being extracted and the chemical properties of the other substances mixed with the organic base being extracted will determine which organic solvent is used.

6. Organic solvent extracts may be dried using a drying agent such as magnesium sulfate or sodium sulfate, if needed.

7. If the organic base being extracted is not volatile, evaporate the solvent under the hood, leaving the extracted organic base. The solvent may also be used for Gas Chromatograph/Mass Spectrometry (GC/MS) analysis if needed, and if appropriate for use in the GC/MS instrument.

8. If the organic base is volatile, or if the salt form of the organic base is desired, add drop wise, the ethyl ether saturated with hydrochloric acid reagent. It is suggested that a check with pH test paper be done to avoid excess hydrochloric acid being added.

9. Evaporate the solvent under the hood and/or filter to isolate the organic salt. Re-crystallization with an organic solvent such as ethyl ether or methanol can be performed if needed.

NOTE: The extractions may be carried out using separatory funnels, test tubes, glass sample vials, beakers, or reaction vessels collected from crime scenes, as needed.

Safety Concerns:

Ethyl ether and other organic solvents are extremely flammable. Sodium Hydroxide and Hydrochloric Acid are caustic and corrosive.

Literature References:

Shriner, R. L., Fuson, R. C., Curtin, D. Y. , **The Systematic Identification of Organic Compounds**, 5th Ed., Wiley and Sons, New York, 1964, pp 67-106.

Moffat, A. C., Ed., **Clarke's Isolation and Identification of Drugs,** 2nd Ed., The Pharmaceutical Press, London, 1986, p. 52.

Berg, E. W., **Physical and Chemical Methods of Separation**, McGraw-Hill, New York, 1963, pp. 6-7.

Adams, R. and J. R. Johnson, **Laboratory Experiments in Organic Chemistry,** 4th Ed., MacMillan, New York, 1949, pp. 103-115.

Canaff, R. F., **A Basic Training Course for Forensic Drug Chemists**, BNDD publication, U. S. Government Printing Office, 1972.

Leffler, J. E., **A Short Course in Modern Organic Chemistry**, MacMillan, New York, 1973, pp. 17-27.

Name of Procedure:

Extractions and Separations
Extraction of Organic Acids

Suggested Uses:

This is a general procedure used to isolate and purify acidic drugs for further analysis.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Pipet with bulb
Graduated cylinder
Glass stirring rod
pH Test paper
Separatory funnel
Funnel
Hydrochloric Acid, concentrated
Sodium Hydroxide
Sodium Sulfate
Magnesium sulfate
Ethyl Ether
Beakers
Filter paper
Chloroform

Formula for Preparing Reagent:

5% Sodium Hydroxide Reagent

1. Weigh out 5 grams of sodium hydroxide.
2. Dissolve in 100 milliliters of water.

3. Pour solution into a reagent bottle.
4. Properly label reagent bottle.

Expiration Date of Reagent:

The reagent can be used until depleted provided it is stored in an airtight reagent bottle.

Application of Procedure on Evidence:

1. Add an amount of sample, equivalent to 5-200 milligrams of the acidic drug to be extracted, to 5-10 milliliters of sodium hydroxide solution in a separatory funnel. The size of the separatory funnel used will depend upon the amount of sample used. Crush and grind any tablet or hard material and remove the powder from capsules before adding to the separatory funnel.

2. Extract the basic solution with two 10-20 milliliter portions of ethyl ether and discard the ether washings.

3. Make the solution in the separatory funnel acidic by adding concentrated hydrochloric acid. Check the pH of the solution with test paper.

4. Extract the acid solution with two 10 milliliter portions of ethyl ether or chloroform. The solvent extracts may be dried using magnesium sulfate or sodium sulfate.

5. Evaporate the solvent to give the free acid compound.

Note: The extractions can be carried out using beakers or test tubes instead of a separatory funnel. For this procedure use glass pipets equipped with rubber bulbs to mix the organic solvents with the aqueous solutions, and to separate the layers. The rest of the procedure is the same in respect to solvent drying, and evaporation.

For more concentrated samples, or for smaller amounts of drugs where only mass spectra data is to be obtained, the separations and sample recovery can be carried out in 3.7 milliliter glass vials.

Safety Concerns:

Ethyl ether is extremely flammable. Sodium hydroxide and hydrochloric acid solutions are caustic and corrosive.

Literature References:

Shriner, R. L., Fuson, R. C., and Curtin, D. Y., **The Systematic Identification of Organic Compounds**, 5th Ed., Wiley and Sons, New York, 1964, pp 67-106.

Moffat, A. C., Ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed., The Pharmaceutical Press, London, 1986, p. 52.

Berg, E. W., **Physical and Chemical Methods of Separation**, McGraw-Hill, New York, 1963, pp. 6-7.

Adams, R., and Johnson, J. R., **Laboratory Experiments in Organic Chemistry**, 4th Ed., MacMillan, New York, 1949, pp. 103-115.

Canaff, R. F., **A Basic Training Course for Forensic Drug Chemists**, BNDD publication, U. S. Government Printing Office, 1972.

Leffler, J. E., **A Short Course in Modern Organic Chemistry**, MacMillan, New York, 1973, pp. 17-27.

Name of Procedure:

Extractions and Separations
Extraction of Volatile Organic Bases

Suggested Uses:

This is a general procedure used to isolate and purify volatile basic drugs for further analysis.

Apparatus Needed to Perform Procedure Including Preparation of the Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Sodium Hydroxide
Sulfuric Acid
Ethyl Ether
Sodium Sulfate, anhydrous
Hydrochloric Acid, concentrated
Conway diffusion dish
Crystallizing dish
Watch glass (or glass plate)
pH Test paper
Reagent bottles
Spatula, small
Glass stirring rod
Beakers
Pipets, glass, disposable
Rubber bulbs
Filter paper
Graduated cylinder

Formula for Preparing Reagent:

20% Sodium Hydroxide Reagent

1. Weigh out 20 grams of sodium hydroxide.
2. Dissolve in 100 milliliters of water.
3. Pour into a reagent bottle.
4. Properly label reagent bottle.

5% Sulfuric Acid Reagent

1. Measure 95 milliliters of water into a 100 milliliter graduated cylinder.
2. Add sulfuric acid to a total volume of 100 milliliters.
3. Pour into a reagent bottle.
4. Properly label reagent bottle.

Ethyl Ether Saturated with Hydrochloric Acid Reagent

1. Place approximately 10 milliliters of concentrated hydrochloric acid into a separatory funnel.
2. Add approximately 50 milliliters of ethyl ether and shake the separatory funnel.
3. Allow the layers to separate.
4. Separate the ethyl ether layer and store in a reagent bottle.
5. Properly label reagent bottle.

Expiration Date of Chemical:

The 20% sodium hydroxide reagent and the 5% sulfuric acid reagent can be used until depleted provided they are stored in an airtight reagent bottle.

The Ethyl ether saturated with hydrochloric acid reagent is prepared as needed and discarded after use.

Application of Procedure on Evidence:

1. Add an amount of sample, equivalent to 5-100 milligrams of the basic drug to 10-20 milliliters of sodium hydroxide reagent in the outer well of a Conway diffusion dish. Crush and grind any tablet or hard material before adding to the well. Place 2-3 milliliters of the sulfuric acid reagent (5%) in the inner well, cover, and place on a warm surface such as a steambath or hot plate.

NOTE: A crystallizing dish fitted with a glass cover and a small beaker can be substituted for the Conway diffusion dish.

2. After 20-30 minutes, remove the dish from the heat source and allow to cool.

3. Transfer the contents of the inner well to a small beaker or test tube and make basic by dropwise addition of sodium hydroxide solution.

4. Extract the basic solution with three 5 milliliter portions of ethyl ether by using a disposable pipet and rubber bulb to mix and separate the organic and liquid phases.

5. The solvent extracts may be dried using magnesium sulfate or sodium sulfate.

6. Add dropwise ether/hydrochloric acid reagent to form the hydrochloride salt, being careful to avoid an excess of acid.

7. If the hydrochloride salt precipitates to produce crystals, collect the crystals on a filter paper, wash with 10-15 milliliters of ether, and allow the crystals to dry on a warm surface. For situations where crystals do not form, evaporate the ether under a stream of nitrogen while applying a moderate amount of heat from a heating device.

Safety Concerns:

Ethyl ether is extremely flammable. Sodium hydroxide and hydrochloric acid are caustic and can cause chemical burns.

Literature References:

Clark, C. C., **"**A Simple Identification Procedure For Some Volatile Amines**"**, **MICROGRAM**, Vol. VI, NO. 5, May 1973, p. 78.

Forensic And Analytical Chemistry of Clandestine Phenethylamines, CND Analytical, Auburn, 1994, p. 8.

Name of Procedure:

Extractions and Separations
Extraction of Amphoteric Compounds Including Morphine, Hydrocodone and other Alkaloids

Suggested Uses:

This is a general extraction procedure used to isolate and purify various amphoteric compounds.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Pipet with bulb
Graduated cylinder
Glass stirring rod
pH Test paper
Separatory funnel
Funnel
Sodium Bicarbonate
Chloroform
Isopropanol
Ethyl Ether
5% Hydrochloric Acid Reagent

Formula for Preparing Reagent:

5% Hydrochloric Acid Reagent

1. Measure 95 milliliters of water in a 100 milliliter graduated cylinder.
2. Bring to total volume (100ml) with concentrated hydrochloric acid.

3. Pour into a reagent bottle.
4. Properly label reagent bottle.

Formula for Preparing Reagent (continued):

3:1 Chloroform/Isopropanol Reagent

1. Measure out 60 milliliters of chloroform.
2. Measure out 20 milliliters of isopropanol.
3. Combine the chloroform and isopropanol.
4. Pour solution into a reagent bottle.
5. Properly label reagent bottle.

Expiration Date of Reagent:

Reagent may be used until depletion if stored In an airtight reagent bottle.

Application of Procedure on Evidence:

1. Dissolve sample in 5% hydrochloric acid reagent.
2. Extract with ethyl ether and discard ether washings.
3. Add sodium bicarbonate until a pH of 8 is obtained. Check with pH test paper.
4. Extract the basic solution with the 3:1 chloroform/isopropanol reagent.
5. The solvent extract may be dried using magnesium sulfate of sodium sulfate.
6. Evaporate the solvent using dry nitrogen and heat to obtain the free base form of the compound.

Safety Concerns:

Ethyl Ether and Isopropanol are flammable solvents. Chloroform should be used in a fume hood.

Literature References:

Shriner, R.L., Fuson, R. C., Curtin, D. Y., **The Systematic Identification of Organic Compounds,** 5th Ed. , Wiley & Sons Inc., New York 1964, pp. 67-85, 88-106.

Butler, William P., **Methods of Analysis**, IRS Publication #341, December 1966, p. 64.

Name of Procedure:

Extractions and Separations
Dry Solvent Extraction of Drugs Utilizing Hexane/Ammonia.

Suggested Uses:

This procedure uses a dry extraction of hexane saturated with ammonia to remove phentermine, propoxyphene, codeine, diethylpropion, diazepam, clorphentermine, amphetamine, ephedrine, phenylpropanolamine, 3,4-methylenedioxyamphetamine and analogs, methamphetamine, clortermine, and meperidine and from pharmaceutical preparations and clandestine mixtures. This procedure also works well in separating organic bases from mixtures containing acetaminophen and nicotinamide.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Ammonium Hydroxide
Hexane
Small beaker
Filter paper
Pipets, glass, disposable
Pipet bulb
Heat source
Reagent bottle

Formula for Preparing Reagent:

1. Place one part ammonium hydroxide and ten parts hexane in a reagent bottle and shake.
2. Allow the layers to separate.
3. Properly label bottle.

Expiration Date of Reagent:

Reagent may be used until depleted if properly stored in an airtight reagent bottle.

Application of Procedure on Evidence:

1. Place 20-50 milligrams of sample in filter paper over a small beaker.
2. Wash sample with ethyl ether and discard washings.
3. Dry sample and then wash with several small portions of hexane/ammonia reagent.
4. Evaporate solvent over moderate heat in a fume hood.

Safety Concerns:

Ammonia is a strong base and is caustic. Ammonia and hexane should be used in a well-ventilated area or under a fume hood.

Literature References:

Adair, A., Noggle, F. Jr., Odom, M., Rhodes, M., "The ANOR (Alternate Non-aqueous Organic Ratio Extraction Procedure)", **MICROGRAM**, Vol. XVI., No. 1, Jan. 1983, pp. 220-224.

Name of Procedure:

Extractions and Separations
Dry Solvent Extraction of Drugs Utilizing Chloroform/Ammonia

Suggested Uses:

This procedure uses a dry extraction of chloroform saturated with ammonia to remove hydromorphone, morphine, diazepam, lorazepam, flurazepam, phentermine, chlordiazepoxide, cocaine, pentazocine, methaqualone, and benzodiazepines from pharmaceutical preparations and clandestine mixtures.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Ammonium Hydroxide
Chloroform
Small beaker
Filter paper
Pipets, glass, disposable
Pipet bulb
Heat source
Reagent bottle

Formula for Preparing Reagent:

1. Mix approximately one part ammonium hydroxide to ten parts chloroform and place in a reagent bottle.
2. Shake reagent bottle and allow layers to separate.
3. Properly label bottle.

Expiration Date of Chemical Reagent:

The reagent can be used until depleted if stored in an airtight reagent bottle.

Application of Procedure on Evidence:

1. Place 20-50 milligrams of sample in filter paper over a small beaker.

2. Wash sample with ethyl ether and discard washings.

3. Dry the sample and then wash with several small portions of chloroform/ammonia reagent.

4. Evaporate solvent over moderate heat in a fume hood.

Safety Concerns:

Ammonia is a strong base and is caustic. Ammonia and chloroform should be used in a well-ventilated area or under a fume hood. Ethyl ether is very flammable.

Literature References:

Adair, A., Noggle, F. Jr., Odom, M., Rhodes, M., "The ANOR (Alternate Non-aqueous Organic Ratio Extraction Procedure)", **MICROGRAM**, Vol. XVI., No. 1, Jan. 1983, pp. 220-224.

Name of Procedure:

Extractions and Separations
Separation of Organic Acids and Bases by Solvent Wash

Suggested Uses:

Organic acids and bases are commonly encountered in mixtures with sugars (mannitol, inositol) or other common diluents. The differences in solubility between the organic acids and bases and these diluent materials can be used to separate the organic components for infrared analysis.

Apparatus Needed to Perform Procedure Including Preparation of Reagents:

Fume hood
Eye protection
Gloves
Laboratory coat
Small beaker
Filter paper
Chloroform
Ethyl Ether
Hexane
Methylene Chloride
Acetone
Spatula, small
Heat source

Application of Procedure on Evidence:

1. Place 10-30 milligrams of sample in filter paper over small beaker.

2. Wash sample with several small portions of suitable solvent.

3. Evaporate solvent over heat source in a fume hood to yield compounds.

Safety Concerns:

Ethyl ether, hexane and acetone are extremely flammable. Chloroform and methylene chloride should be used in a well ventilated area or in a fume hood.

Other:

This procedure can also be used to remove controlled substances from commercial preparations.

Examples: Diazepam can be removed from tablets with an acetone or ethyl ether wash of the crushed tablet and methylphenidate can be removed using chloroform.

Literature References:

Forensic and Analytical Chemistry of Clandestine Phenethylamines, CND Analytical, Inc., 1994, pp. 6-7.

Moffat, A. C. Ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed., The Pharmaceutical Press, London 1986.

Name of Procedure:

Extractions and Separations
Extraction of Psilocybe Mushrooms

Suggested Uses:

This procedure is used to extract psilocybe mushrooms.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Eye protection
Gloves
Laboratory coat
Small beaker
Pipets, glass, disposable
Pipet bulb
Glass stirring rod
pH Test paper
Test tube
Test tube rack
Clean glass vial with cap
Glacial Acetic Acid
Ammonium Hydroxide
Ethyl Ether
Nitrogen source

Formula for Preparing Reagent:

3:1 Chloroform/Isopropanol Reagent

1. Measure out 60 milliliters of chloroform.
2. Measure out 20 milliliters of isopropanol.
3. Combine the chloroform and isopropanol.
4. Pour solution into a reagent bottle.

5. Properly label reagent bottle.

Application of Procedure on Evidence:

1. Break up approximately 1-2 grams of psilocybe mushrooms and place in a small beaker.

2. Add enough deionized water to moisten.

3. Add 1-2 milliliters of glacial acetic acid and check with test paper to assure that the solution is acidic.

4. Stir 1-2 minutes. **[Do not leave in acidic solution for extended period of time]**

5. Decant liquid to test tube.

6. Add concentrated ammonium hydroxide dropwise until a pH of 8 is obtained.

7. Gently extract with ethyl ether or 3:1 chloroform/isopropanol.

8. Solvent may be dried using magnesium sulfate of sodium sulfate.

9. Evaporate solvent under dry nitrogen.

Safety Concerns:

Ethyl ether is extremely flammable. Ammonium hydroxide is a strong base and glacial acetic acid is a strong acid. Care should be taken to keep these two components capped when not in use and away from each other to avoid mixing.

Literature References:

Casale, J., "An Aqueous-Organic Extraction Method for the Isolation and Identification of Psilocin from Hallucinogenic Mushrooms", **Journal of Forensic Science**, January

1985.

Modified by Chemist T.H. McSwain with the North Carolina State Bureau of Investigation Drug Chemistry Laboratory, in use in the laboratory since January, 1985.

Name of Procedure:

Extractions and Separations
Extraction of Anabolic Steroids from Vegetable Oils

Suggested Uses:

This procedure is used to isolate anabolic steroids from various vegetable oil preparations.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Eye protections
Laboratory coat
Gloves
Methanol
Heptane
Hexane
Reagent bottles
Disposable syringe with needle
Beakers
Pipets, glass, disposable
Rubber bulbs
Heat source
Nitrogen gas source
Centrifuge, Hamilton Bell, Vanguard V6000
Centrifuge tubes, 15ml polypropylene

Application of Procedure on Evidence:

1. Withdraw 1 milliliter of oil using a syringe and transfer the oil to a centrifuge tube.

2. Add 2 milliliters of heptane or hexane and mix well.

3. Add 1 milliliter of methanol and mix well.

4. Centrifuge for 2-3 minutes to separate the layers.

5. Transfer the methanol layer to a small beaker and evaporate methanol using a steam bath or warm heating device and nitrogen.

Safety Concerns:

Methanol, hexane and heptane are flammable.

Literature References:

Chiong, D. M., Consuegra-Rodrigues, E., Almirall, J. R., "The Analysis and Identification of Steroids", **Journal of Forensic Science**, Vol, 37, No. 37, March, 1992, pp. 488-502.

Clark, C. C., "The GLC Quantitation of Some Anabolic Steroids in Vegetable Oil Preparations", **MICROGRAM**, Vol. XXV, No. 10, October 1992, pp. 255-268.

Name of Procedure:

Extractions and Separations
Separation of Cocaine Base and Benzocaine Base

Suggested Uses:

This procedure is used to separate cocaine base and benzocaine base.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Hydrochloric Acid Solution (0.05N)
Ethyl Ether
Concentrated Sodium Hydroxide
Test tube
Pipets, glass, disposable
Pipet bulb
Small beaker
pH Test paper
Sodium Sulfate, anhydrous
Filter paper
Heat source

Formula for Preparing Reagent:

0.05N Hydrochloric Acid Reagent

1. Measure out 1 milliliter of concentrated hydrochloric acid.
2. Add the hydrochloric acid to 250 milliliters of water.
3. Place the solution in a reagent bottle.
4. Properly label reagent bottle.

Application of Procedure on Evidence:

1. Crush 20-50 milligrams of sample and place in test tube.

2. Add approximately 2-3 milliliters of hydrochloric acid solution.

3. Vortex approximately 30 seconds and quickly remove liquid to second test tube. [Do not allow sample to remain in acid for extended period of time.]

4. Extract aqueous layer several times with ethyl ether and discard. [If infrared of benzocaine is desired, evaporate ethyl ether.]

5. Make aqueous layer basic with sodium hydroxide. Check with test paper for pH10.

6. Extract with ethyl ether and dry through sodium sulfate in filter paper.

7. Evaporate ethyl ether over moderate heat to obtain cocaine base.

Safety Concerns:

Ethyl ether is extremely flammable. Hydrochloric acid is corrosive. Sodium hydroxide is caustic.

Literature References:

Analytical Profiles of Cocaine, Local Anesthetics and Common Diluents Found With Cocaine, CND Analytical, Inc. 1990, p.11.

Name of Procedure:

Extractions and Separations
Separation of Cocaine Base and Procaine Base

Suggested Uses:

This procedure is primarily used to separate cocaine base and procaine base. Common diluents such as, benzocaine, levamisole, and other hexane insoluble compounds can also be removed by utilizing this procedure.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Heat source
Eye protection
Laboratory coat
Gloves
Beaker or sample vial
Pipette(s) with bulb
Test tube(s)
Vortex mixer (optional)
Hexane
Water
Methylene Chloride (optional)
Sodium Sulfate, anhydrous (optional)

Application of Procedure on Evidence:

1. Crush a portion of the sample and place in test tube. Size of the portion used will be dictated by the ratio of diluents(s) to cocaine base present in the sample. (Suggested size is at least 20-30 milligrams if available.)

2. Add approximately 2 milliliters hexane to test tube.

3. Vortex or agitate test tube (optional.)

4. Allow layers to separate and any insoluble material to settle to the bottom.

Application of Procedure on Evidence (continued):

5. Remove hexane to second test tube (optional).

6. Add approximately 10 milliliters water to hexane. (Fill rest of test tube.)

7. Vortex or agitate test tube (optional).

8. Remove hexane layer and evaporate hexane over moderate heat to obtain sample. (A drying agent such as sodium sulfate may be used prior to evaporating hexane.)

9. If a large amount of procaine or other diluent(s) are present in the sample, additional washings of the hexane can be performed prior to evaporation. Remove the hexane layer to a new test tube and add additional aliquots of water. Repeat as necessary.

Safety Concerns:

Keep top of test tubes pointed away from face or covered while vortexing to avoid splashing in eyes or face.

Other:

If an infrared of procaine or other diluent(s) is/are desired, extract the water solution with approximately 25 milliliters of methylene chloride. A drying agent such as sodium sulfate may be used in filter paper if needed.

Literature References:

Kerr, K., “A Simple Procedure for Separating Cocaine Base from Procaine Base”, **MICROGRAM**, Vol. XXIII, NO. 5, MAY 1990, pp. 93-96.

Name of Procedure:

Extractions and Separations
Separation of Cocaine Hydrochloride and Dimethylterephthalate

Suggested Uses:

This procedure is used to separate mixtures of cocaine hydrochloride and dimethylterephthalate in order to isolate cocaine base.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
5% Hydrochloric Acid Reagent
Ethyl Ether
Sodium Hydroxide
Small beaker
pH Test paper
Reagent bottles
Spatula, small
Glass stirring rod
Heat source

Formulation for Preparing Reagent:

5% Hydrochloric Acid Reagent

1. Measure 95 milliliters of water in a 100 milliliter graduated cylinder.
2. Bring to total volume (100ml) with concentrated hydrochloric acid.
3. Pour into a reagent bottle.
4. Properly label reagent bottle.

Application of Procedure on Evidence:

1. Dissolve 10-20 milligrams of cocaine/DMT mixture in 5% hydrochloric acid reagent.
2. Wash with ethyl ether 2-3 times and discard ether (unless IR of DMT is desired).
3. Add sodium hydroxide to aqueous solution and check for pH 10 with test paper.
4. Extract with ethyl ether.
5. Evaporate ethyl ether over moderate heat to obtain sample.

Safety Concerns:

Ethyl ether is flammable. Sodium hydroxide is caustic and can cause chemical burns. Hydrochloric acid is corrosive. This procedure should be performed in a well-ventilated area or in a fume hood.

Literature References:

Moffat, A.C., Ed., **Clarke's Isolation and Identification of Drugs** , 2nd Ed., The Pharmaceutical Press, London 1986.

Shriner, R. L., Fuson, R. C., Curtin, D. Y., **The Systematic Identification of Organic Compounds**, 5th Ed., Wiley & Sons Inc., New York 1964, pp. 67-85, 88-106.

Name of Procedure:

Extractions and Separation
Separation of Cocaine Hydrochloride and Lidocaine Hydrochloride

Suggested Uses:

This procedure is used to separate mixtures of cocaine hydrochloride and lidocaine hydrochloride in order to isolate cocaine for infrared analysis. Various sugars and other water soluble diluents do not interfere with the separation.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Acetone
Ethyl ether
Sodium Sulfate, anhydrous
Sodium Bicarbonate
pH Test paper
Reagent bottles
Spatulas, small
Glass stirring rods
Filter paper
Small beakers

Application of Procedure on Evidence:

1. Place 10-200 milligrams of powder on filter paper over a beaker and wash sample with 5-15 milliliters of acetone. If no diluents are present in the sample, remove cocaine hydrochloride from the filter paper for further analysis.

2. For samples diluted with sugars or other related material, place the filter paper

over another beaker and wash the residue through with 5 milliliters of water.

3. Add sodium bicarbonate to the filtrate until the solution is basic and extract with 5-10 milliliters of ethyl ether. Evaporate ether to give cocaine base.

Safety Concerns:

Acetone and ethyl ether are extremely flammable.

Literature References:

Shriner, R. L., Fuson, R. C., Curtin, D. Y., **The Systematic Identification of Organic Compounds,** 5th Ed., Wiley and Sons, New York, 1964, pp. 67-85.

Name of Procedure:

Extractions and Separations
Separation of Dextropropoxyphene Napsylate and Acetaminophen

Suggested Uses:

This procedure is used to separate dextropropoxyphene napsylate from acetaminophen for further analysis.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Concentrated Sodium Hydroxide Solution (cold/refrigerated)
Ethyl Ether
Sodium Sulfate, anhydrous
Filter paper
Small beaker
Glass stirring rod
Clean glass vial with cap
Nitrogen source

Application of Procedure on Evidence:

1. Remove film coating from tablet if one is present.

2. Crush tablet and place in small beaker.

3. Add 2-3 drops of cold, concentrated sodium hydroxide solution and stir until all powder is wet and a paste is formed.

5. Wash with ethyl ether and dry through sodium sulfate in filter paper.

6. Evaporate ethyl ether under nitrogen.

Safety Concerns:

Sodium hydroxide is caustic and ethyl ether is extremely flammable. This procedure should be performed in a well-ventilated area or in a fume hood.

Literature References:

Stall, W., "Separation of Darvon and APC", **MICROGRAM**, Vol. XI, NO. 11, Nov. 1978, pp. 204-206.

Name of Procedure:

Extractions and Separations
Extraction of Diethylpropion from a Time-release Preparation

Suggested Uses:

This procedure is used to isolate diethylpropion from a time-release tablet.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Hydrochloric Acid
Ethyl Ether
Sodium Sulfate, anhydrous
pH Test paper
Volumetric flask, 10-25ml
Reagent bottles
Spatulas, small
Scalpel and blade
Glass stirring rods
Beakers
Pipets, glass, disposable
Rubber bulbs
Filter paper
Centrifuge, Hamilton Bell, Vanguard V6000
Centrifuge tubes, 15ml polypropylene
Sodium Hydroxide Solution, concentrated

Formula for Preparing Reagent:

5% Hydrochloric Acid Reagent

1. Measure 95 milliliters of water in a 100 milliliter graduated cylinder.
2. Bring to total volume (100ml) with concentrated hydrochloric acid.
3. Pour into a reagent bottle.
4. Properly label reagent bottle.

Formula for Preparing Reagent (continued):

Ethyl Ether saturated with Hydrochloric Acid Reagent

1. Place approximately 10 milliliters of concentrated hydrochloric acid into a separatory funnel.
2. Add approximately 50 milliliters of ethyl ether and shake the separatory funnel.
3. Allow the layers to separate.
4. Separate the ethyl ether layer and store in a reagent bottle.
5. Properly label reagent bottle.

Expiration Date of Chemical:

5% Hydrochloric acid reagent may be used until depletion if stored in an airtight reagent bottle.

Ethyl ether saturated hydrochloric acid reagent is prepared as needed and discarded after use.

Application of Procedure on Evidence:

1. Crush and grind tablet (or portion), after first removing film coating with a scalpel if necessary, and then transfer 200-300 milligrams of the powder to a centrifuge tube.

2. Mix the powder well with 10 milliliter of hydrochloric acid solution, centrifuge for 3-5 minutes, and

3. Extract with ethyl ether and discard washings.

4. Make the solution basic by adding sodium hydroxide solution, checking pH with test paper.

5. Extract the basic solution with ethyl ether.

6. Ethyl ether extracts may be dried using magnesium sulfate or sodium sulfate.

7. Add ether/hydrochloric acid solution to precipitate diethylpropion hydrochloride, being careful to avoid an excess of hydrochloric acid. This can be accomplished by careful drop-wise addition of the acidic ether.

Application of Procedure on Evidence (continued):

8. The diethylpropion hydrochloride can be recovered for further analysis by evaporating the ether using heat and nitrogen, or by collecting the crystals on filter paper and washing with ether.

Safety Concerns:

Ethyl ether is extremely flammable. Sodium hydroxide and hydrochloric acid solution are caustic and corrosive.

Literature References:

Adapted by Chemist J. R. Daniel, N. C. State Bureau of Investigation, Raleigh, 1992.

Shriner, R. L., Fuson, R. C., Curtin, D. Y., **The Systematic Identification of Organic Compounds**, 5th Ed., Wiley and Sons, New York, 1964, pp.88-106.

Moffat, A. C., Ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed., The Pharmaceutical Press, London, 1986, p. 52.

Name of Procedure:

Extractions and Separations
Extraction of Lysergic Acid Diethylamide (LSD)

Suggested Uses:

This procedure is used to extract LSD from capsules, liquid, tablets or blotter paper.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Sodium Bicarbonate
Chloroform
pH Test paper
Sodium Sulfate, anhydrous
Pipets, glass, disposable
Pipet bulb
UV Light source

Application of Procedure on Evidence:

1. Dissolve sample in water.

2. Add sodium bicarbonate until pH is 8. Check with test paper.

3. Extract with chloroform and evaporate over heat source for further analysis.

Safety Concerns:

Chloroform should be used in a well ventilated area or in a fume hood. LSD should not come into contact with skin.

Literature References:

Zacharias, D., "Infrared Identification of LSD in Capsules, Liquid, Sugar Cubes and Tablets", **MICROGRAM**, Vol. I, No. 4, Jan. 1968, pp. 23-24.

Name of Procedure:

Extractions and Separations
Extraction of Lysergic Acid Diethylamide (LSD) From Sugar Cubes

Suggested Uses:

This procedure is used to extract LSD from sugar cubes.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Eye protection
Gloves
Laboratory coat
Spot plate
Methanol
Micropipets, glass, disposable
Glass vial with cap
UV Light source

Application of Procedure on Evidence:

1. Place sugar cube under UV light and find the most concentrated spot of fluorescence. Hold this side closest to spot well.

2. Wash cube with methanol dropwise over spot well.

3. Collect concentrated methanol with micropipette as it evaporates around edge of spot well.

4. Transfer concentrated methanol to glass vial. The sample is now ready for further analysis.

Safety Concerns:

Methanol is a flammable organic solvent. LSD should not come into contact with skin.

Literature References:

Moffat, A.C., Ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed., The Pharmaceutical Press, London 1986, p. 52.

Name of Procedure:

Extractions and Separations
Separation of Methamphetamine Hydrochloride and Dimethyl Sulfone

Suggested Uses:

This procedure is used to separate methamphetamine and dimethyl sulfone in order to identify methamphetamine hydrochloride.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Eye protection
Gloves
Laboratory coat
Ethyl Ether
Acetone
Chloroform
Methanol
Small beaker
Filter paper
Steam bath or other heat source

Application of Procedure on Evidence:

1. Place 10-20 milligrams of methamphetamine/dimethyl sulfone sample in a piece of filter paper over a small beaker.

2. Wash mixture with ethyl ether and discard used solvent.

3. Wash mixture with acetone and discard used solvent (unless infrared of dimethyl sulfone is desired.)

4. Wash mixture with chloroform and evaporate solvent over heat source, yielding the methamphetamine hydrochloride.

Safety Concerns:

Ethyl ether and acetone are extremely flammable solvents. Chloroform should be used in a well ventilated area or under a fume hood.

Other:

Methamphetamine can be further purified using methanol and ethyl ether.

Literature References:

Moriwaki, W. and Lee, M., "Analytical Note Dimethyl Sulfone in Methamphetamine Exhibits", **MICROGRAM**, Vol. XXIX, No. 3, March 1996, pp. 58-60.

NOTE: This article states that the methamphetamine is present in the acetone layer. Actually it is present in the chloroform layer.

Name of Procedure:

Extractions and Separations
Separation of Cocaine Hydrochloride and Nicotinamide

Suggested Uses:

This procedure is used to separate mixtures of cocaine hydrochloride and nicotinamide in order to isolate cocaine base. The procedure can be applied to mixtures of the two compounds that also contain other water soluble materials.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Ethyl Ether
Sodium Hydroxide
Sodium Sulfate, anhydrous
Reagent bottles
Pipets, glass, disposable
Rubber bulbs
Spatulas, small
Beaker
Filter paper
Centrifuge, Hamilton Bell, Vanguard V6000
Centrifuge tubes, 15ml polypropylene
Magnesium Sulfate

Formula for Preparing Reagent:

5% Sodium Hydroxide Reagent

1. Weigh out 5 grams of sodium hydroxide.

2. Dissolve in 100 milliliters of water.
3. Pour solution into a reagent bottle.
4. Properly label reagent bottle.

Expiration Date of Chemical Reagent:

Reagent may be used until depletion if it is properly stored in an airtight reagent bottle.

Application of Procedure on Evidence:

1. Place 50-100 milligrams of powdered sample into a centrifuge tube and add 7-8 milliliters of water.

2. Add one drop of sodium hydroxide reagent, mix well, and centrifuge for 1-2 minutes.

3. Remove aqueous layer by using a disposable pipet or by decanting, leaving a solid material in the bottom of the centrifuge tube.

4. Wash the solid material with 2-3 milliliters of water and discard the washing.

5. Extract the solid material in the centrifuge tube with 2-3 milliliters of ethyl ether and filter the ether into a beaker. Repeat this extraction as needed.

6. Dry the ethyl ether using sodium sulfate or magnesium sulfate.

7. Evaporate the ethyl ether, yielding cocaine base.

Safety Concerns:

Sodium hydroxide is caustic and can cause chemical burns. Ethyl ether is extremely flammable.

Literature References:

Shriner, R. L., Fuson, R. C., Curtin, D. Y., **The Systematic Identification of Organic Compounds**, 5th Ed., Wiley and Sons, New york, 1964, pp. 67-85.

Name of Procedure:

Extractions and Separations
Separation of Cocaine from Procaine, Benzocaine, Dimethlyterephthalate, and Nicotinamide.

Suggested Uses:

This procedure separates cocaine from some diluents including, but not limited to: procaine, benzocaine, dimethylterephthalate, and nicotinamide.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Gloves
Eye protection
Laboratory coat
Acetone
Ethyl ether
Sodium bicarbonate
(+) Di-*p*-toluoyl-D-tartaric acid, anhydrous (TLTA)
pH Test paper
Reagent bottles
Spatula, small
Filter paper
Test tubes, medium
Beakers, small

Formula for Preparing Reagent:

50 mg/mL TLTA Reagent

1. Dissolve 1.0 gram TLTA in 20 milliliters of acetone in a small reagent bottle.

2. Properly label the reagent bottle.

Expiration Date of Reagent:

The 50 mg/mL TLTA reagent can be used until depleted provided it is stored in an airtight bottle.

Application of Procedure on Evidence:

1. Dissolve 10-20 milligrams of a cocaine base sample in 2 milliliters of acetone in a test tube, or make cocaine HCl basic with sodium bicarbonate paste and add acetone.

2. Filter the mixture through fluted filter paper and catch filtrate in a clean test tube.

3. Add 10-12 drops of TLTA Reagent to the filtrate and invert the test tube to mix.

4. If a precipitate does not form, go to step 6.

5. If a precipitate does form, allow the mixture to sit for several minutes. Then repeat steps 2 & 3 until no precipitate forms when the TLTA reagent is added to the filtrate.

6. Crystallize the cocaine TLTA by scratching the inside of the test tube.

7. Allow the mixture to sit for several minutes. Filter the mixture through fluted filter paper, wash the cocaine TLTA with small portions of acetone and discard the acetone.

8. Convert the cocaine TLTA to cocaine base using a sodium bicarbonate solution, and extract the cocaine base with ethyl ether. Evaporate the ether to obtain cocaine base.

Safety Concerns:

Acetone and ethyl ether are extremely flammable.

Literature References:

Ard, Edwina., Jackson Police Crime Laboratory, Jackson, MS, "Clean-up of Contaminated 'Crack' Procedure", presented at the Southern Association of Forensic Scientists Fall Meeting in September 1990, in Jacksonville, FL.

Additional Literature References:

The below listed references were referred to by Edwina Ard in her paper "Clean-up of Contaminated 'Crack' Procedure". These references are listed for information purposes:

Almirall, Jose R. and Hanlon, Christopher, "Purification and Analysis of Benzocaine Contaminated 'Rocks'," **Microgram**, Vol. XXI, No.10 (October 1988) pp.174-177.

Ard, Edwina, "Separation of Phencyclidine from Crude Reaction Mixtures", **Microgram**, Vol. XX, No. 10 (October 1987), pp. 186-189.

Galka, Karen L., "Separation of Cocaine from Benzocaine", **Microgram**, Vol. XXIII, No. 5 (May 1990), pp. 97-98.

Hussain, Anwar, "A Simple Method for the Separation and Identification of Cocaine and Lidocaine in a Mixture", **Microgram,** Vol. XXI, No. 6 (June 1988) pp. 100-101.

Kerr, Keith, "A Simple Procedure for Separating Cocaine Base from Procaine Base", **Microgram**, Vol. XXIII, No. 5 (May 1990), pp. 93-96.

Kessler, Robert R., "Separation of Cocaine from Common Adulterants and Diluents", **Microgram**, Vol. XVIII, No. 10 (October 1984), pp. 149-150.

Saba, Haytham, "Separation and Identification of Cocaine Base and Ephedrine Base", **Microgram**, Vol. XXII, No. 1 (January 1989), pp. 8-9.

Saloom, Joseph M., "Separation of Cocaine from Impure 'Crack'," **Microgram**, Vol. XXII, No. 2 (February 1989), pp. 22-30.

Sorgen, Gary J., "Purification of Cocaine by Salt Formation", **Microgram**, Vol. X, No. 4 (April 1977), pp. 52-56.

Sorgen, Gary J., "Identification of *d*- and *l*- Cocaine", **Microgram**, Vol. XVI, No. 8 (August 1983), pp. 126-131.

Sorgen, Gary J. and Heagy, James A., "Extraction of Cocaine from Currency", **Microgram**, Vol. XVI, No. 8 (August 1983), pp. 132-133.

Name of Procedure:

Extractions and Separations
Separation of Cocaine Base and Cinnamoyl Cocaines

Suggested Uses:

This procedure is used to remove cinnamoyl cocaines from cocaine base. Additionally this procedure will remove nicotinamide, procaine, caffeine, stearic acid, sodium bicarbonate, sodium borate, and "field test blue". This procedure is less efficient at removing anhydroecgonine methyl ester and will not remove methylbenzoate which appear similar to cinnamoyl cocaines by infrared analysis.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Eye protection
Gloves
Laboratory coat
Hexane
Test tube
Pipets, glass, disposable
Pipet bulb
Vortex mixer
Small beaker
Heat source
Graduated cylinder
50ml beaker
Glass stirring rod
Potassium permanganate
Funnel
Reagent bottle
Spatula
Water

Formula for Preparing Reagent:

Note: This is the same reagent as used in Procedure A-6 for Potassium Permanganate Color Test.

1. Weigh out 0.3 gram of potassium permanganate.

2. Dissolve in 30 milliliters water.

3. Pour into a reagent bottle.

4. Properly label reagent bottle.

Quality Control Check:

A quality control check of this reagent will be performed using a known standard of a barbiturate and following the Procedure A-6.

Expiration Date of Chemical Reagent:

No expiration date. Reagents need to be properly contained in a sealed container and stored in a cool place.

Application of Procedure on Evidence:

1. Crush 30 milligrams of sample and place in test tube.

2. Add 3 milliliters hexane to tube and vortex 30 seconds.

3. Add 1 milliliter of potassium permanganate reagent and vortex 1 minute.

4. Squirt approximately 10 milliliters of deionized water through mixture.

5. Allow layers to separate.

6. Remove hexane layer.

7. Evaporate hexane over moderate heat to obtain cocaine base.

Safety Concerns:

Keep top of test tubes pointed away from face or covered while vortexing to avoid splashing in eyes or face.

Literature References:

Kerr, K., "A Simple Procedure for Separating Cocaine Base from Procaine Base", **MICROGRAM**, Vol. XXIII, NO. 5, MAY 1990, pp. 93-94.

Moffat, A. C., ed., **Clarke's Isolation and Identification of Drugs**, Pharmaceutical Press, London, 1986, p. 1170.

Casale, J. F. and Klein, R. F. X. "Illicit Production of Cocaine", **Forensic Sci Rev**, 5:95, 1993.

Name of Procedure:

Extractions and Separations
Electrophilic Separation of Cocaine Base

Suggested Uses:

This procedure is used to remove lidocaine base from cocaine base. Additionally this procedure removes nicotinamide, procaine, caffeine, stearic acid, sodium bicarbonate, sodium borate, carbohydrates, and “field test blue”.

Apparatus Needed to Perform Procedure Including Preparation of Reagent:

Fume hood
Eye protection
Gloves
Laboratory coat
Hexane
Test tube
Pipets, glass, disposable
Pipet bulb
Vortex mixer
Small beaker
Heat source
50mL beaker
Glass stirring rod
Sulfuric acid (concentrated)
Trioxane (trioxymethylene)
Funnel
Reagent bottle
Spatula
Water
Sodium hydroxide
pH paper

Formula for Preparing Reagent:

Marquis Reagent

Note: This is the same reagent as used in Procedure A-1 for Marquis Color Test.

1. Place 10 milliliters of concentrated sulfuric acid in beaker.
2. Add 8-10 drops of formaldehyde solution (40%) and stir.
3. Pour solution into reagent bottle.
4. Properly label reagent bottle.

Alternate Method

1. Pour 15-20 milliliters of concentrated sulfuric acid into a reagent bottle.
2. Add 0.2-0.3 gram of trioxane (trioxymethylene) and stir until completely dissolved.
3. Properly label reagent bottle.

20% Sodium Hydroxide Reagent

1. Weigh out 20 grams of sodium hydroxide.
2. Dissolve in 100 milliliters of water.
3. Pour into reagent bottle.
4. Properly label reagent bottle.

Quality Control Check:

A quality control check of the Marquis reagent will be performed using a known standard of heroin and following procedure A-1.

Expiration Date of Chemical Reagent:

The Marquis reagent should be prepared every 30 days.

Application of Procedure on Evidence:

1. Crush 0.2 - 0.3 gram of sample and place in test tube.
2. Add 3 milliliters of hexane to tube and vortex 30 seconds.
3. Add 5 drops of Marquis reagent and vortex 1 minute.
4. Add 20% sodium hydroxide reagent with mixing until mixture is basic to pH paper.
5. Squirt approximately 10 milliliters of deionized water through mixture.
6. Allow layers to separate.
7. Remove hexane layer.
8. Evaporate hexane over moderate heat to obtain cocaine base.

Safety Concerns:

Keep top of test tubes pointed away from face or covered while vortexing to avoid splashing in eyes or face. Sulfuric acid and sodium hydroxide are caustic and can cause chemical burns.

Literature References:

Kerr, K., "A Simple Procedure for Separating Cocaine Base from Procaine Base", **Microgram**, Vol. XXIII, No. 5, May 1990, pp. 93-94.

Moffat, A. C., ed., **Clarke's Isolation and Identification of Drugs**, 2nd Ed., Pharmacuetical Press, London, 1986, p. 139-140.

Gould, E. S., **Mechanism and Structure in Organic Chemistry**, Holt, Rinehart, and Winston, Inc., 1959, 428-436.

Name of Procedure:

Extractions and Separations
Extraction of Psilocybe Mushrooms #2

Suggested Uses:

This procedure is used to extract psilocybe mushrooms.

Apparatus Needed to Perform Procedure:

Fume hood
Eye protection
Gloves
Laboratory coat
Mortar and pestle
Deionized water
Sodium bicarbonate
Ethyl ether
Chloroform or methanol
Glass vial

Application of Procedure on Evidence:

1. Grind approximately 1 gram of psilocybe mushrooms with a mortar and pestle.

2. Add water and sodium bicarbonate (approximately 10 grams) until an off-white paste forms.

3. Add ether and stir.

4. Decant off ether.

5. Evaporate solvent.

6. Reconstitute in chloroform or methanol to inject on GC/MS.

Safety Concerns:

Ethyl ether is extremely flammable.

Literature References:

Drug Enforcement Administration Special Testing and Research Laboratory, Forensic Chemist Seminar, February 6-10, 2006.